

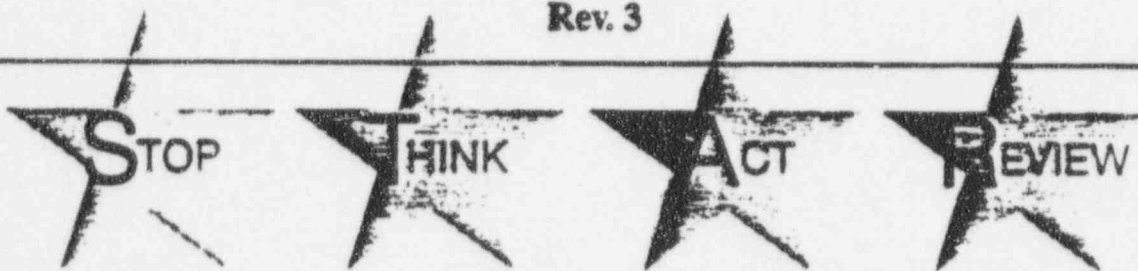
MILLSTONE NUCLEAR POWER STATION  
CHEMISTRY PROCEDURE



Routine Operation and Calibration of the  
Laboratory Ion Chromatography Systems

CP 801/2801/3801Y

Rev. 3



Approval:

David Paff  
for Sr. Vice President - Millstone Station

SORC Mtg. No: Biennial Review Date: 3-8-96

Effective Date: 1-3-94

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Millstone All Units  
Chemistry Procedure

Routine Operation and Calibration of the Laboratory Ion Chromatography Systems

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ATTACHMENTS AND FORMS

Chem Form 801/2801/3801Y-1, "Millstone Chemistry Laboratory Method Log For Ion Chromatograph"

Chem Form 801/2801/3801Y-2, "Method Development For Ion Chromatograph"

Chem Form 801/2801/3801Y-3, "Millstone Chemistry Laboratory Ion Chromatograph Control Standard Log"

Chem Form 801/2801/3801Y-4, "Millstone Chemistry Department Ion Chromatograph Raw Data Log"

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## 1. PURPOSE

### 1.1 Objective

Provide instruction for routine operation of Dionex Ion Chromatographs including:

- Instrument start-up
- Analysis of samples
- Instrument calibration
- Data manipulation
- Method development

### 1.2 Discussion

The general term "chromatography" means the separation of components from a complex mixture by a technique based on selective absorption. All chromatography methods employ a mobile phase and a stationary phase to separate the analyte(s) of interest. The basic principle behind "Ion Chromatography" is the specific rate of ion exchange that exists between the sample ion and the counter-ion (eluent or mobile phase) with the stationary phase (resin). This rate of ion exchange determines the time necessary for the sample ion to pass through the separator (analytical) resin column and be detected.

### 1.3 Frequency

1.3.1 When in use Ion Chromatographs are calibrated weekly, or when a control standard indicates a problem with existing calibration.

1.3.2 When in use a control standard is run daily.

## 2. PREREQUISITES

### 2.1 General

2.1.1 Power available (115 volts) to instrument.

2.1.2 Valve actuating gas at 80 - 120 psi delivery pressure.

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## 2.2 Documents

2.2.1 Control Chart

2.2.2 When specified by Chemistry management, Instrument Performance Trend Chart.

2.2.3 IC notebook

## 2.3 Tools and Consumables

2.3.1 Reagents, standards, eluents, and regenerates have been prepared.

2.3.2 High purity water, Nanopure or Super Q.

## 2.4 Definitions

- ACI – Advanced Computer Interface
- CIM – Computer Interface Module
- IC – Ion Chromatograph

### NOTE

The following definitions relate to the operation of the Process 450 Software which is windows based.

- **DOUBLE CLICK** – When using a computer mouse, this means to press the left button twice in rapid succession. This will open a computer file.
- **SINGLE CLICK** – When using a computer mouse, this means to press the left button once. This is used to select a computer file, menu, or command.
- **SINGLE CLICK-RIGHT** – When using a computer mouse, this means to press the right button once.
- **HOLD CLICK** – When using a computer mouse, this means to press and hold the left button and not to release it until the desired task is accomplished.



- DRAG – When using a computer mouse, this means to move a computer generated image from one position to another.  
For example – HOLD CLICK and DRAG Process 450 icon to bottom of CRT screen.

### 3. PRECAUTIONS

- 3.1 Bottles containing eluent and regenerate should not be pressurized beyond 15 psi as the bottles may explode causing serious injury to laboratory personnel.
- 3.2 Samples containing elevated levels (greater than 1.0 ppm) of hydrazine, metals, or organics should not be analyzed as damage to the resin columns and suppressor system may occur.
- 3.3 Care should be taken when tightening plastic fittings as to avoid damaging plastic threads. A torque wrench should be used if a torque value is specified.
- 3.4 Spilled or leaked eluents and regenerates should be cleaned up as soon as possible to avoid damage to system components.
- 3.5 Except during initial startup, eluent pumps should not be operated without first setting minimum and maximum pressure settings as damage may occur to the pumps if these settings are not set.
- 3.6 Care should be taken when working around system plumbing to ensure that gas lines cannot become kinked, punctured or otherwise damaged.

#### 4. INSTRUCTIONS

##### NOTE

1. The methods that have been created for Ion Chromatography analyses are listed on Chem Form 801/2801/3801Y-1, "Millstone Chemistry Laboratory Method Log For Ion Chromatograph." Method parameters are identified in Chem Form 801/2801/3801Y-2, "Method Development For Ion Chromatograph." Each method will have its own completed Method Development Form. This form should be reviewed if there are any questions regarding execution of the subject method. New methods can be created following the guidance provided in Sec. on 4.12.
2. Names given in parenthesis in action steps are the software program given names to computer function keys as displayed in the CRT.

##### 4.1 Procedure Entry Position

4.1.1 IF running IC AND the operating software is Auto Ion 300, Go To Section 4.2.

4.1.2 IF running IC AND operating software is Process 450<sup>or Peaknet</sup> for IBM compatible computers, Go To Section 4.7. chg #1

4.1.3 IF developing a method for IC, Go To Section 4.12.

##### 4.2 Start-Up Autoion 300 Software

4.2.1 Refer To Chem Form 801/2801/3801Y-2 and VERIFY the following is consistent with method to be run:

- Eluent
- Regenerant
- Plumbing
- Resin columns
- Suppressor
- Injection source: loop, or concentrator column

4.2.2 ENSURE the following:

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- There is an adequate supply of regenerate and eluent to complete required analyses.
- Valve actuating gas is at desired delivery pressure.

4.2.3 IF IC has been turned off, PRESS power buttons to each component as follows:

### NOTE

The "On" button for the Aution 300 data station (computer) is located on the back of the CRT. The computer will automatically load the operating software from the hard disk.

- Printer
- Chromatography Module
- Computer Interface Module
- Computer

4.2.4 VERIFY the following components are in "REMOTE" position:

- Analytical Pump Module
- Conductivity Detector Module
- Chromatography Module

4.2.5 WHEN computer has finished booting up, PRESS "ENTER" to continue.

4.2.6 PRESS "K7."

4.2.7 Using the keyboard, SET date.

4.2.8 PRESS "ENTER."

4.2.9 PRESS "K9."

4.2.10 Using the keyboard, SET time.

4.2.11 PRESS "ENTER."

- 4.2.12 WHEN date and time have been set, PRESS "K5" (CONTINUE) twice.
- 4.2.13 ENTER CIM address of 705.
- 4.2.14 PRESS "ENTER."
- 4.2.15 ENTER CIM default configuration file name.
- 4.2.16 PRESS "ENTER."
- 4.2.17 PRESS "K5" (CONTINUED) to display main menu.
- 4.2.18 PRESS "KO" (EXECUTE) to advance to auto-monitor page.

### 4.3 Running a Sample-Aution 300 Software

#### NOTE

Methods are run from the auto-monitor page displayed on the CRT. Function keys are displayed on the bottom of the screen. The first step of running a method is to load the method into the CIM. After the CIM is loaded, the method is initiated. Information defining method parameters such as system number and CIM address can be found on Chem Form 801/2801/3801Y-2, "Method Development For Ion Chromatograph" for method being run. Unit 3's software differs slightly from Unit 1's as the exact number and order of computer prompts is not identical.

- 4.3.1 PRESS "K2" (LOAD CIM).
- 4.3.2 WHEN prompted, Refer To Chem Form 801/2801/3801Y-2 and ENTER response as follows:

PROMPT	RESPONSE
CIM address number?	705 or 706
System 1 or 2?	1 or 2
Use schedule?	N
Name of method file?	Method name
Sample name?	Sample name
Any additional prompts	Default value

- 4.3.3 PLACE sample inlet tube in sample bottle.
- 4.3.4 PRESS "K7" (START).

## NOTE

The analysis will automatically start and run through to completion after the system number has been entered into computer.

- 4.3.5 WHEN prompted, Refer To Chem Form 801/2801/3801Y-2, and ENTER response as follows:

<u>PROMPT</u>	<u>RESPONSE</u>
CIM address number?	705 or 706
System number?	1 or 2

- 4.3.6 WHEN sample analysis has finished, PERFORM the following:

- REVIEW chromatogram for proper integration of peaks.
- IF any peaks are poorly integrated, Refer To Section 4.6, "Manual Integration of Peaks."
- RECORD data and INITIAL  
Chem Form 801/2801/3801Y-4.
- REMOVE sample inlet tube from sample bottle and PLACE  
in bottle containing DI water.

## 4.4 Calibration of IC - Aution 300 Software

- 4.4.1 Before calibrating instrument, RUN at least one blank sample of Nanopure or Super Q water to insure that:

- Baseline is stable.
- There is no contamination in system.
- No leaks in system.
- The sample intake pumps are properly primed and are delivering the expected sample volume.

- 4.4.2 VERIFY that auto-monitor page is displayed on CRT.

- 4.4.3 PRESS "K2" (LOAD CIM).

## NOTE

The calibration information is automatically updated when the sample name is entered as "AUTOCAL #." The "#" part of the name corresponds to the number of the standard as it relates to a multi-standard calibration curve. For example, if one standard was used to calibrate the instrument, the standard name would be "AUTOCAL." If 2 standards were used to define the calibration curve, the names of the standards would be "AUTOCAL1," and "AUTOCAL2." If a third standard was used, its name would be "AUTOCAL3," and etc.

- 4.4.4 WHEN prompted, Refer To Chem Form 801/2801/3801Y-2, and ENTER response as follows:

<u>PROMPT</u>	<u>RESPONSE</u>
CIM address number?	705 or 706
System 1 or 2?	1 or 2
Use schedule?	N
Name of method file?	Method name
Sample name?	AUTOCAL #
Any additional prompts	Default

- 4.4.5 PLACE sample inlet tube in standard to be analyzed.

- 4.4.6 PRESS "K7" (START).

- 4.4.7 WHEN prompted, Refer To Chem Form 801/2801/3801Y-2, and ENTER response as follows:

<u>PROMPT</u>	<u>RESPONSE</u>
CIM address number?	705 or 706
System number?	1 or 2

- 4.4.8 WHEN standard analysis is finished, PERFORM the following:
- COMPARE area or height of standard with previous standard run.
  - IF difference exceeds plus or minus 5.0%, INVESTIGATE reason for difference.
  - REVIEW chromatogram for proper integration of peaks.
  - NOTIFY Chemistry supervision of any identified problems.
  - IF no further analyses are to be performed on calibration standard, REMOVE sample inlet tube from standard bottle and place in storage bottle containing DI water.



#### 4.5 Analysis of Control Standard – Autoion 300 Software

4.5.1 **VERIFY** that auto-monitor page is displayed on CRT.

4.5.2 **PRESS** “K2” (LOAD CIM).

4.5.3 **WHEN** prompted, Refer To Chem Form 801/2801/3801Y-2, and **ENTER** response as follows:

<u>PROMPT</u>	<u>RESPONSE</u>
CIM address number?	705 or 706
System 1 or 2?	1 or 2
Use schedule?	N
Name of method file?	Method name
Sample name?	CTRL
Any additional prompts	Default

4.5.4 **PLACE** sample inlet tube in control standard to be analyzed.

4.5.5 **PRESS** “K7” (START).

4.5.6 **WHEN** prompted, Refer To Chem Form 801/2801/3801Y-2, and **ENTER** response as follows:

<u>PROMPT</u>	<u>RESPONSE</u>
CIM address number?	705 or 706
System number?	1 or 2

4.5.7 **WHEN** control standard analysis is finished, **PERFORM** the following:

- a. **RECORD** data and **INITIAL**  
Chem Form 801/2801/3801Y-3.
- b. Refer To control chart and **PLOT** result.
- c. **IF** trend chart is available, Refer To trend chart and **PLOT** value.
- d. **REVIEW** chromatogram for proper integration.
- e. **IF** control standard value is outside of acceptance criteria, **INVESTIGATE** problem and **NOTIFY** Chemistry supervision.

f. IF applicable, PROVIDE brief explanation of problem and follow-up actions on the following:

- control chart
- Chem Form 801/2801/3801Y-3

#### 4.6 Manual Integration of Peaks - Autoion 300 Software

### NOTE

The following instruction is for integrating the last sample to be analyzed by the IC. This is the only chromatogram that can be reprocessed if the data is not stored. The cursor is moved left or right by rotating the "wheel" on the keyboard. The cursor is moved up and down by hitting the "shift" key, then rotating the wheel.

- 4.6.1 VERIFY that auto-monitor page is displayed on CRT.
- 4.6.2 PRESS "K4" (REPROCESS DATA).
- 4.6.3 PRESS "K5" (REPLOT).
- 4.6.4 MOVE cursor to a position on chromatogram that is slightly below and in front of area of interest.
- 4.6.5 PRESS "KO" (ANCHOR).
- 4.6.6 MOVE cursor to form box surrounding area of interest.
- 4.6.7 PRESS "K1" (ZOOM).
- 4.6.8 MOVE cursor to beginning of peak to be integrated.
- 4.6.9 PRESS "K7" (PEAK START).
- 4.6.10 MOVE cursor to end of peak to be integrated.
- 4.6.11 PRESS "K8" (PEAK AREA/HEIGHT).
- 4.6.12 PRESS "K6" (REPORT RESULT).
- 4.6.13 PRESS "K3" (HARDCOPY).
- 4.6.14 PRESS "K4" (REPROCESS DATA)

4.6.15 IF additional peaks are to be re-integrated, Go To step 4.6.3.

4.6.16 PRESS "KO" (AUTO-MONITOR).

#### 4.7 Start-Up Process 450 Software

4.7.1 Refer To Chem Form 801/2801/3801Y-2 and **VERIFY** following is consistent with method to be run:

- Eluent
- Regenerant
- Plumbing
- Resin columns
- Suppressor
- Injection source, loop, or concentrator column

4.7.2 **ENSURE** the following:

- There is an adequate supply of regenerate and eluent to complete required analyses.
- Valve actuating gas is at desired delivery pressure.

#### NOTE

Dionex Series 450 will automatically boot up to the main menu upon power-up.

4.7.3 IF IC has been turned off, PRESS power buttons to each component as follows:

- a. Printer
- b. Chromatography Module
- c. Advanced Computer Interface Module
- d. Compute



4.7.4 VERIFY the following components are in "REMOTE" position:

- Analytical Pump Module
- Conductivity Detector Module
- Chromatography Module

4.7.5 LOCATE "Analyze" icon.

4.7.6 DOUBLE CLICK "Analyze" icon to open program.

4.7.7 SINGLE CLICK "File" pull down menu.

4.7.8 SINGLE CLICK "Monitor."

#### NOTE

The "Run" icon and "Trend" icon will appear at the bottom left corner of the screen when a analyzer has been selected and approved.

4.7.9 SELECT analyzer to be opened and SINGLE CLICK.

4.7.10 SINGLE CLICK "OK."

4.7.11 To select additional analyzers, Go To 4.7.7.

4.7.12 MOVE mouse arrow to down arrow located in top right corner of analyze window and SINGLE CLICK to reduce window to icon.

4.7.13 HOLD CLICK and DRAG "Process 450" main menu icon to bottom of screen.

#### 4.8 Running a Sample – Process 450 Software or Peaknet Software

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##### NOTE

Samples are analyzed using the "Run Program." This program can monitor (real time) multiple systems simultaneously. The analyzers selected under the Analyze Program will have their own "child" system windows (cation and anion) displayed when the run icon is opened. The systems are selected by clicking their title bar, which highlights the bar in blue. When the ACI starts sampling for the system (i.e., a method is running) a real time chromatogram is displayed in the system window; the status of the system is shown at the bottom of the window.

- 4.8.1 PLACE sample inlet tube in sample to be analyzed.
- 4.8.2 IF "Run Window" is not displayed on CRT, DOUBLE CLICK "Run" icon to display window.
- 4.8.3 SINGLE CLICK system title bar "Anion" or "Cation" at top of system window to select system to be run.
- 4.8.4 SINGLE CLICK "Load" pull down menu located at top of "Run Window."
- 4.8.5 SINGLE CLICK "Method" from pull down menu.
- 4.8.6 SELECT method to be run and SINGLE CLICK.
- 4.8.7 IF correct method has been selected, SINGLE CLICK "OK."
- 4.8.8 SINGLE CLICK "Run" pull down menu located at top of run Window.
- 4.8.9 SINGLE CLICK "Start" from pull down menu.

##### NOTE

For trending purposes, samples from the same system should always be named exactly the same.

- 4.8.10 ENTER sample name inside sample box.
- 4.8.11 SINGLE CLICK "OK."

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4.8.12 WHEN sample analysis has finished, PERFORM the following:

- a. REVIEW chromatogram for proper integration of peaks.
- b. IF any peaks are poorly integrated, Refer To Section 4.11, "Manual Integration of Peak – Process 450 Software."
- c. RECORD data and INITIAL Chem Form 801/2801/3801Y-4. or Peaknet Software
- d. REMOVE sample inlet tube from sample bottle and PLACE in bottle containing DI water.

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4.9 Calibration of IC – Process 450 Software or Peaknet Software

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### NOTE

The calibration information is automatically updated when the sample name is entered as "AUTOCAL #." The "#" part of the name corresponds to the number of the standard as it relates to a multi-standard calibration curve. For example, if one standard was used to calibrate the instrument, the standard name would be "AUTOCAL." If 2 standards were used to define the calibration curve, the names of the standards would be "AUTOCAL1," and "AUTOCAL2." If a third standard was used, its name would be "AUTOCAL3," and etc.

4.9.1 Before calibrating instrument, RUN at least one blank sample of Nanopure or Super Q water to insure that:

- Baseline is stable.
- There is no contamination in system.
- No leaks in system.
- The sample pumps are properly primed and are delivering the expected sample volume.

4.9.2 PLACE sample inlet tube in standard to be analyzed.

4.9.3 VERIFY that "Run Window," auto-monitor, for system to be calibrated is displayed on the CRT.

4.9.4 SINGLE CLICK system title bar "Anion" or "Cation" at top of system window to select system to be run.

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- 4.9.5 SINGLE CLICK "Load" pull down menu located at top of "Run Window."
- 4.9.6 SINGLE CLICK "Method" from pull down menu.
- 4.9.7 SELECT method to be run and SINGLE CLICK
- 4.9.8 SINGLE CLICK "OK."
- 4.9.9 SINGLE CLICK "Run" pull down menu located at top of "Run Window."
- 4.9.10 SINGLE CLICK "Start" from pull down menu.
- 4.9.11 ENTER standard name inside sample box.
- 4.9.12 SINGLE CLICK "OK."
- 4.9.13 WHEN standard analysis has finished, PERFORM the following:
  - a. COMPARE area or height of standard with previous standard run.
  - b. IF difference exceeds plus or minus 5.0%, INVESTIGATE reason for difference.
  - c. REVIEW chromatogram for proper integration of peaks.
  - d. NOTIFY Chemistry supervision for any identified problems.
  - e. IF no further analyses are to be performed on calibration standard, REMOVE sample inlet tube from standard bottle and PLACE in storage bottle containing DI water.

4.10 Analysis of Control Standard - Process 450 Software or *Paulnet Software* <sup>chp</sup>

- 4.10.1 PLACE sample inlet tube in control standard to be analyzed.
- 4.10.2 SINGLE CLICK system title bar "Anion" or "Cation" at top of system window to select system to be run.
- 4.10.3 SINGLE CLICK "Load" pull down menu located at top of Run Window.
- 4.10.4 SINGLE CLICK "Method" from pull down menu.

- 4.10.5 SELECT method to be run and SINGLE CLICK.
- 4.10.6 SINGLE CLICK "OK."
- 4.10.7 SINGLE CLICK "Run" pull down menu located at top of "Run Window."
- 4.10.8 SINGLE CLICK "Start" from pull down menu.

### NOTE

For trending purposes, samples from the same system should always be named exactly the same.

- 4.10.9 ENTER control standard name inside sample box.
- 4.10.10 SINGLE CLICK "OK."
- 4.10.11 WHEN control standard analysis is finished, PERFORM the following:
  - a. RECORD data and INITIAL  
Chem Form 801/2801/3801Y-3.
  - b. Refer To control chart and PLOT result.
  - c. IF trend chart is available, Refer To trend chart and PLOT value.
  - d. REVIEW chromatogram for proper integration.
  - e. IF control standard value is outside of acceptance criteria, INVESTIGATE problem and NOTIFY Chemistry supervision.
  - f. PROVIDE brief explanation of problem and follow-up actions on the following:
    - control chart
    - Chem Form 801/2801/3801Y-3
  - g. REMOVE sample inlet tube from calibration standard and place in bottle containing DI water.

#### 4.11 Manual Integration of Peak – Process 450 Software or Peaknet Software

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##### NOTE

Chromatograms can be re-integrated from the Optimize Program. This program can be used to fine tune the integration, assign peak names and to update method files and data files to include new parameters. This section deals with "Set Baseline Manually" file of the Optimize Program, which only affects the chromatogram that is being worked on.

4.11.1 DOUBLE CLICK "~~Process 450~~" main menu icon.

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4.11.2 DOUBLE CLICK "Optimize" icon.

4.11.3 SINGLE CLICK "File" pull down menu.

4.11.4 SINGLE CLICK "Open Data File."

##### NOTE

The list of file directories is located at the bottom left of window.

4.11.5 SELECT raw data file directory that contains chromatogram that is to be reprocessed.

4.11.6 SINGLE CLICK "OK."

4.11.7 SELECT data file name of chromatogram.

4.11.8 SINGLE CLICK "OK."

4.11.9 SINGLE CLICK "Operations" pull down menu.

4.11.10 SINGLE CLICK "Set Baseline Manually."

4.11.11 SELECT peak to be re-integrated by one of the following methods:

- In box located in top left hand corner, SINGLE CLICK peak number that is to be edited.
- SINGLE CLICK-RIGHT peak on displayed chromatogram.

4.11.12 SINGLE CLICK "View" menu.

4.11.13 SINGLE CLICK "Start/Stop" markers option.

4.11.14 MOVE mouse cursor to a position that is slightly below and in front of area of interest.

### NOTE

The mouse cursor can be used to zoom in on the peak to be integrated. This makes it very easy to see where a peak should begin and end. The scale tool box, located in the upper right hand corner of the baseline window, can also be used to enlarge and concentrate on the area of interest. The whole chromatogram can be moved by using the arrow keys (up, down, left and right) that border the chromatogram.

4.11.15 HOLD CLICK and MOVE cursor until area of interest is contained in highlighted box.

4.11.16 RELEASE mouse button.

4.11.17 MOVE cursor along baseline of chromatogram in front of peak until double arrow appears.

4.11.18 HOLD CLICK and MOVE cursor to position on chromatogram where peak should start.

4.11.19 RELEASE mouse button.

4.11.20 MOVE cursor along baseline of chromatogram in back of peak until double arrow appears.

4.11.21 HOLD CLICK and MOVE cursor to position on chromatogram where peak should end.

4.11.22 RELEASE button.

4.11.23 WHEN desired changes have been made, SINGLE CLICK "File" pull down menu.

4.11.24 SINGLE CLICK "View Report."

4.11.25 IF satisfied with report, SINGLE CLICK "Print."

4.11.26 SINGLE CLICK "OK."

- 4.11.27 SINGLE CLICK bar located in top left corner of baseline window.
- 4.11.28 SINGLE CLICK "Close."
- 4.11.29 SINGLE CLICK bar located in top left hand corner of "Optimize" window.
- 4.11.30 SINGLE CLICK "Close."
- 4.11.31 IF you want to save desired changes, SINGLE CLICK "Yes"
- 4.11.32 IF you *do not* want to save changes, SINGLE CLICK "No" to leave data as is.
- 4.11.33 IF "Yes" was selected, SINGLE CLICK "OK."
- 4.11.34 SINGLE CLICK bar in upper left hand corner of "~~Process 450~~" window. *chg #1*
- 4.11.35 SINGLE CLICK "Minimize" to reduce window to icon.

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Chemistry  
Technician, or  
Chemistry  
Specialist

#### 4.12 Method Development

### NOTE

Chem Form 801/2801/3801Y-2 is the Method Development Form for the Ion Chromatograph. Completion of this form will document all the analytical parameters of the IC method being developed.

- 4.12.1 IF developing a method for Ion Chromatograph, COMPLETE Chem Form 801/2801/3801Y-2 as follows:
- a. RECORD following information in Section A:
- Name of method
  - Name of person(s) developing method
  - Name of chemist that reviewed method

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- b. RECORD following information in Section B:
- Analytes to be detected
  - Matrix of sample
  - General description of analytical range
- c. RECORD following information in Section C:
- Instrument unit number
  - System number of IC, 1 or 2
  - CIM address
- d. RECORD following information in Section D:
- Description of separator column
  - Description of guard column
  - Concentrator column or sample loop
- e. RECORD description of suppressor system in Section E.
- f. IF desired, RECORD schematic of plumbing system in Section F.
- g. RECORD instruction on how to prepare eluent in Section G including the following:
- Typical flow rate
  - Expected conductivity
- h. RECORD instruction on how to prepare regenerate in Section H.
- i. RECORD instruction on how to prepare standard in Section I and COMPLETE calibration and control standard tables.



j. RECORD following calibration data in Section J:

- Analytical range of method.
- Number of calibration standards used to define calibration curve.
- Type of curve fit.
- Limit of detection and limit of quantification.

4.12.2 ATTACH following to completed Chem Form 801/2801/3801Y-2:

- Computer hardcopy of IC method.
- Calibration data, which includes graph and chromatograms.
- Examples of typical sample chromatograms.

4.12.3 IF not a chemist, SEND completed form to chemist for review.

Chemist →

4.12.4 REVIEW method, especially preparation of standards and reagents.

4.12.5 PLACE signature on method development form indicating approval of method.

4.12.6 RETURN form to method developer.

Chemist,  
Chemistry  
Technician, or  
Chemistry  
Specialist →

4.12.7 COPY method development form and attachments.

4.12.8 PLACE original in IC notebook and SEND copy to Analytical Chemist.

4.12.9 RECORD method data on Chem Form 801/2801/3801Y-1.

Level of Use  
Information

STOP

THINK

ACT

REVIEW

CP 801/2801/3801Y  
Rev. 3  
22 of 24

#### 4.13 Start-Up Process Peaknet Software

- 4.13.1 ENSURE there is an adequate supply of regenerate and eluent to complete required analysis.

#### NOTE

Dionex Series Peaknet will automatically boot up to the main menu upon power-up.

- 4.13.2 IF IC has been turned off, PRESS power button to each component as follows:

- a. Printer
- b. Chromatography module
- c. Conductivity detector
- d. Computer
- e. Gradient pump module

- 4.13.3 Using mouse, LOCATE and DOUBLE CLICK on "Dionex Peaknet" icon.

- 4.13.4 Using mouse, LOCATE and DOUBLE CLICK on "Main Menu" icon.



## 5. REVIEW AND SIGNOFF

5.1 The review and signoff for this procedure is located on the following Chemistry forms:

- Chem Form 801/2801/3801Y-1
- Chem Form 801/2801/3801Y-2
- Chem Form 801/2801/3801Y-3
- Chem Form 801/2801/3801Y-4

## 6. REFERENCES

6.1 Dionex Aution 300 Data Station Operator's Manual, Dionex Corporation, Sunny Vale, CA

6.2 AI-450 Chromatography Software User's Guide, Dionex Corporation, Sunny Vale, CA

6.3 Peaknet Chromatography Software User's Guide,  
7. SUMMARY OF CHANGES Dionex Corp., Sunny Vale, CA.

Chg  
#1

7.1 Change calibration frequency from daily to weekly, step 1.3.1.

7.2 Added instruction concerning the following:

- How to manually integrate a peak on a chromatogram, Section 4.6.
- How to operate "Process 450 Software" in the following Sections:
  - 4.7
  - 4.8
  - 4.9
  - 4.10
  - 4.11
- How to complete Chem Form 801/2801/3801Y-2, "Method Development For Ion Chromatograph," Section 4.12

Level of Use  
Information

STOP

THINK

ACT

REVIEW

CP 801/2801/3801Y  
Rev. 3  
23 of 24

- 7.3 The content of this procedure was modified to incorporate new format criteria specified in Revision 0 of the "Millstone Procedure Writer's Guide."

Lot 374

1-3-94

93-54

Millstone Chemistry Laboratory  
Method Log For Ion Chromatograph

[illegible]

**Method Development For Ion Chromatograph**

A. Method Name: \_\_\_\_\_  
\_\_\_\_\_

Developed By: \_\_\_\_\_ / \_\_\_\_\_  
(Print) (Signature)

Reviewed By: \_\_\_\_\_ / \_\_\_\_\_  
(Print) (Signature)

B. Objective of Method: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

C. Instrument Identification: \_\_\_\_\_  
(Unit, System, CIM)

D. Columns: Separator: \_\_\_\_\_  
Guard: \_\_\_\_\_  
Concentrator: \_\_\_\_\_

E. Suppressor: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_



F. Plumbing Description/Schematic:  
(Optional)

G. Eluent  
Preparation:

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Flow Rate: \_\_\_\_\_

Conductivity: \_\_\_\_\_

H. Regenerate:  
Preparation:

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Flow Rate: \_\_\_\_\_

I. Standards:

Calibration Standards			
Chemical Specie	Concentration 1	Concentration 2*	Concentration 3*
1)			
2)			
3)			
4)			
5)			
6)			

\*Optional

Control Standards			
Chemical Specie	Concentration 1	Concentration 2*	Concentration 3*
1)			
2)			
3)			
4)			
5)			
6)			

\*Acceptable deviation should be determined by statistical analysis.

Preparation:

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J. Calibration Data:

1. Range of Method: \_\_\_\_\_
2. Number of Standards Used: \_\_\_\_\_
3. Type of Curve Fit: \_\_\_\_\_
4. LOD: \_\_\_\_\_
5. LOQ: \_\_\_\_\_

Form Approved by Sr. Vice President - Millstone Station

Effective Date

SOAC Mtg. No.

**Milestone Chemistry Laboratory**  
**Ion Chromatograph Control Standard Log**

[illegible]

Comments:

SOBC Mtg. No.

98-54

Millstone Chemistry Department  
Ion Chromatograph Raw Data Log

RANG 3

[illegible]



John Delf  
Form Approved by Sr. Vice President - Millstone Station

1-3-94  
Effective Date

93-54  
SORC Mtg. No.

**Method Development For Ion Chromatograph**

A. Method Name: CATION 1

Developed By: BREWER / CRONE  
(Print)

[Signature]  
(Signature)

Reviewed By: BWS Giff  
(Print)

[Signature]  
(Signature)

B. Objective of Method: ANALYSIS OF LOW LEVEL SODIUM  
AND PPM LEVEL ETA FOR SECONDARY  
WATER SAMPLES, USING sample  
concentration and an isocratic  
run.

C. Instrument Identification: UNIT 2 ACI 1 SYS 1  
(Unit, System, CIM)

D. Columns: Separator: CS 12  
Guard: CG 12  
Concentrator: CG 12

E. Suppressor: CATION SRS  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

I. Standards:

Calibration Standards			
Chemical Specie	Concentration 1	Concentration 2*	Concentration 3*
1) SODIUM	.5 ppb	5.0 ppb	10.0 ppb
2) ETHANOLAMINE	500 ppb	2500 ppb	5000 ppb
3)			
4)			
5)			
6)			

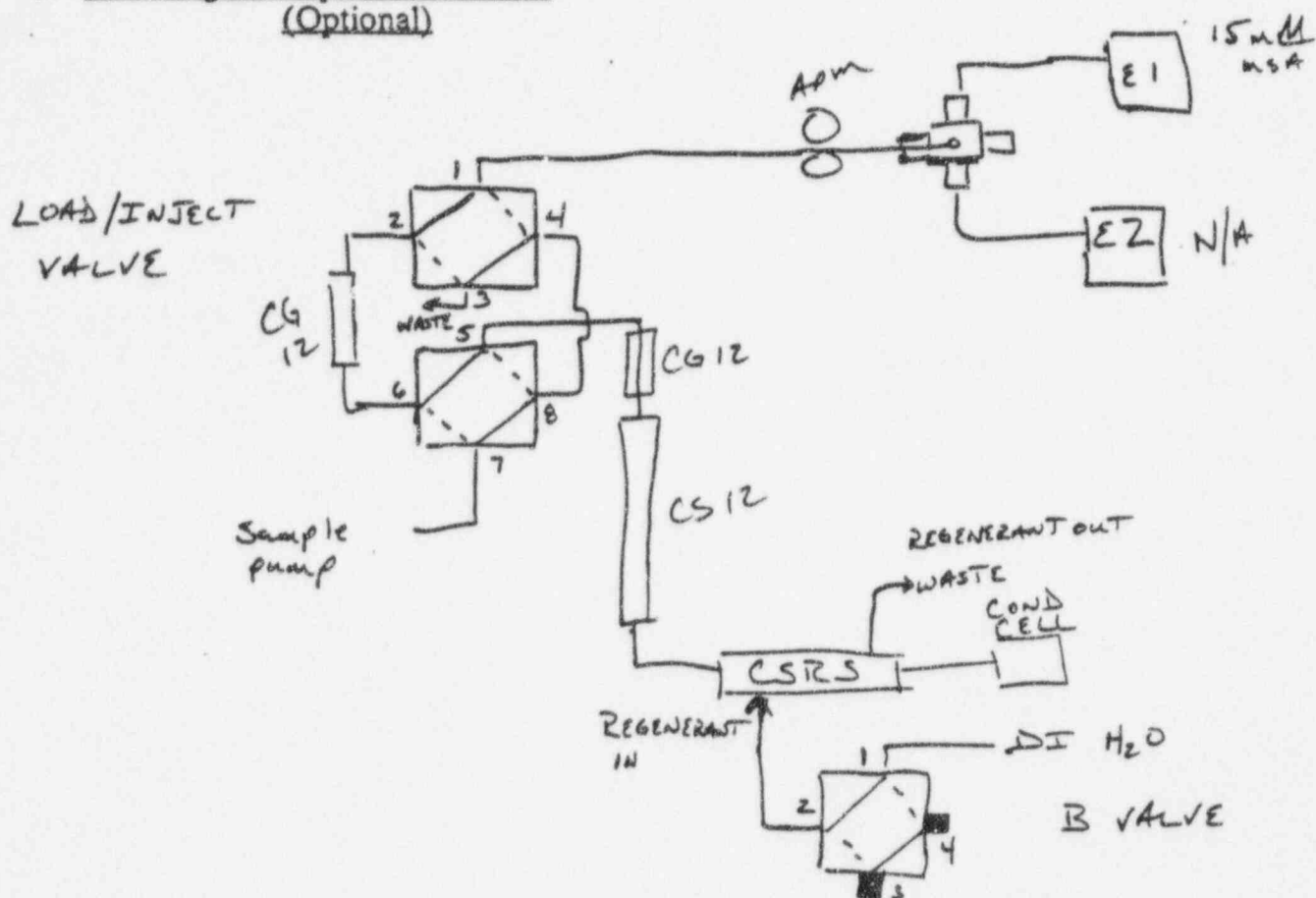
\*Optional

Control Standards			
Chemical Specie	Concentration 1	Concentration 2*	Concentration 3*
1) SODIUM	2.0 ppb		
2) ETHANOLAMINE	3.0 ppm		
3)			
4)			
5)			
6)			

\*Acceptable deviation should be determined by statistical analysis.

Preparation: SODIUM AND ETA ARE PREPARED THROUGH  
DILUTIONS OF STOCK SOLUTIONS  
eg CONTROL STD 200ml of 1 ppm Na<sup>+</sup>  
and 300ml of 1000 ppm ETA diluted to 100ml

F. Plumbing Description/Schematic:  
(Optional)



G. Eluent  
Preparation:

15 mM MSA DILUTE 60 ml of 1.0 M  
MSA TO 4.0 liter with DI water

Flow Rate:

1.0 ml/min

Conductivity:

1.0 mS

H. Regenerate:  
Preparation:

CATION SRS VOLTAGE SET at 2  
using DI WATER for REGENERANT

Flow Rate:

N/A

J. Calibration Data:

1. Range of Method: Na<sup>+</sup> .1 ppb - 10 ppb ETA 50 ppb - 5 to 10 ppm
2. Number of Standards Used: 3 (zero for AUTOCAL 1)
3. Type of Curve Fit: QUADRATIC
4. LOD: Na - .013 ~~.05~~ ETA < 50 ppb
5. LOQ: Na - .05 ppb ETA 50 ppb

—

# Method Development For Ion Chromatograph

A. Method Name: ETA MET

Developed By: BREWER / CRONE  
(Print)

[Signature]  
(Signature)

Reviewed By: Bob Galt  
(Print)

[Signature]  
(Signature)

B. Objective of Method: ANALYSIS OF HIGH LEVEL ETHANOLAMINE  
USING LOOP INJECTION AND AN  
ISOCRATIC RUN.

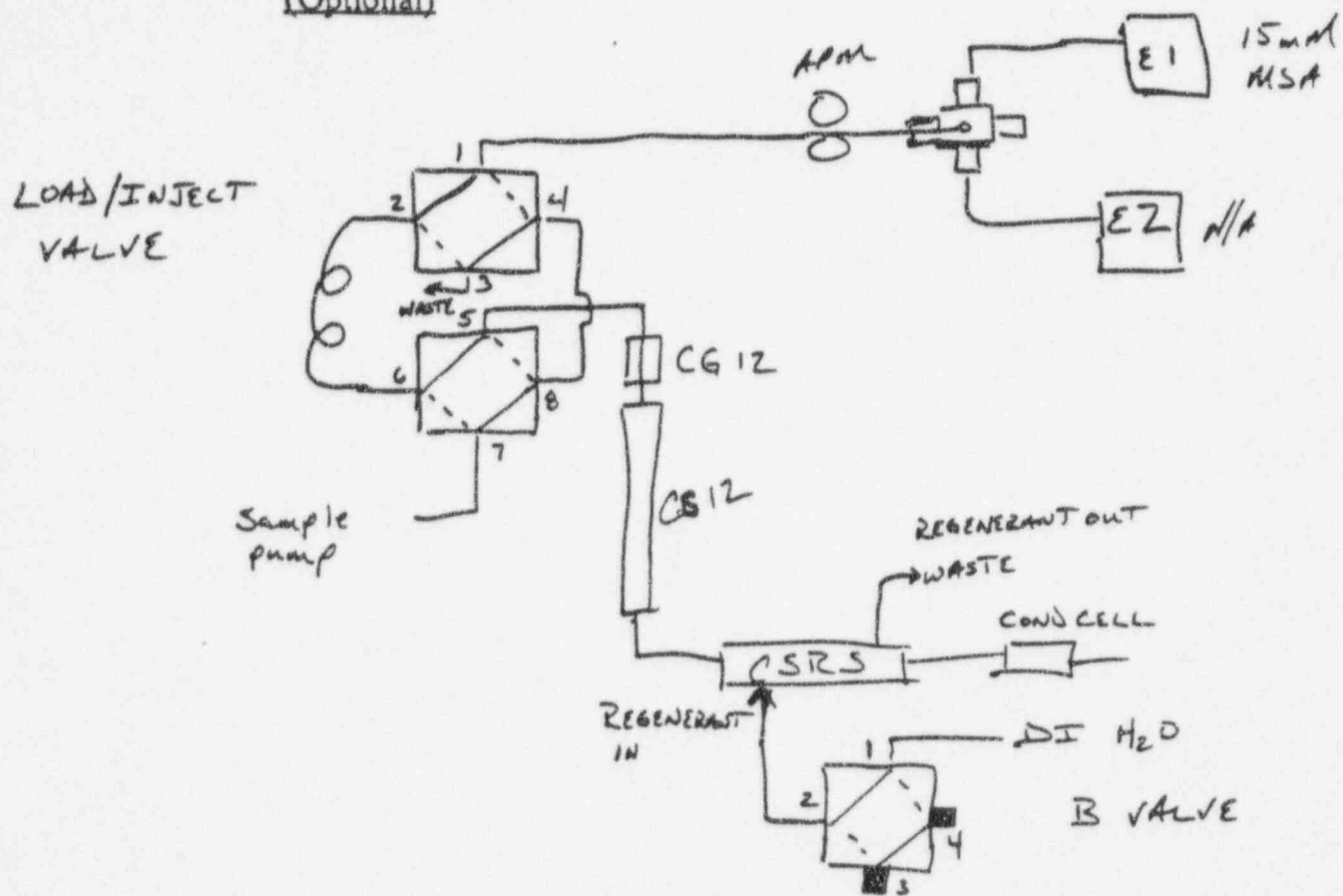
C. Instrument Identification: UNIT 2 ACT 1 SYS 1  
(Unit, System, CIM)

D. Columns: Separator: PS 12  
Guard: HA CG 12  
Concentrator: N/A

E. Suppressor: ATION SRS



F. Plumbing Description/Schematic:  
(Optional)



G. Eluent

Preparation:

15 mM MSA DILUTE 60 ml of 1.0 M  
MSA TO 4.0 liter with DI water

Flow Rate:

1.0 ml/min

Conductivity:

1.0 mS

H. Regenerate:

Preparation:

CATION SRS VOLTAGE SET at 2  
using DI WATER for REGENERANT

Flow Rate:

n/a

NOTE: AMMONIA ADDED

6-8-95

Note: 10 ppm std added on 9/3/94 to increase range of analysis

I. Standards:

Calibration Standards			
Chemical Specie	Concentration 1	Concentration 2*	Concentration 3*
1) ETA	.5 ppm	2.5 ppm	10 ppm
2) Ammonia	0.05 ppm	0.1 ppm	0.2 ppm
3)			
4)			
5)			
6)			

\*Optional

Control Standards			
Chemical Specie	Concentration 1	Concentration 2*	Concentration 3*
1) ETA	1.0 ppm		
2) Ammonia	0.075 ppm		
3)			
4)			
5)			
6)			

\*Acceptable deviation should be determined by statistical analysis.

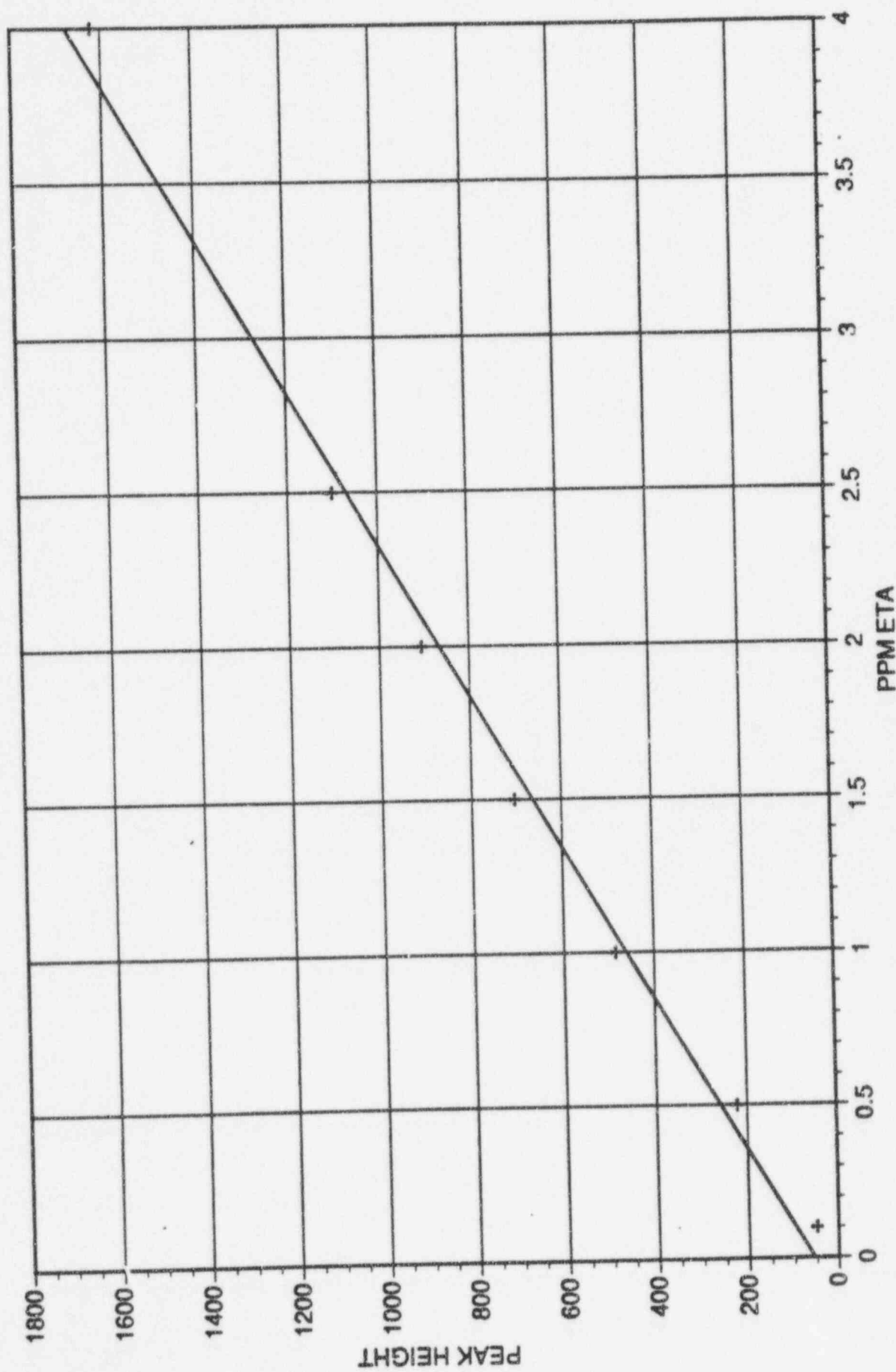
Preparation: FROM 1000 ppm STOCK ETA SOLUTION DILUTE

AS FOLLOWS TO 100 ml with DI WATER			
1000 ppm ETA STOCK	1000 ppm ETA STD	10 ppm NH <sub>4</sub> STOCK	10 ppm NH <sub>4</sub> STD
50 ml	.5 ppm	500 ml	50 ppb
250 ml	2.5 ppm	1000 ml	100 ppb
1000 ml	1.0 ppm	2000 ml	200 ppb
1.0 ml	10 ppm	750 ml	75 ppb

J. Calibration Data:

- 10 ppm
1. Range of Method: .05 ppm - 4 ppm <sup>9/3/94</sup> RI
2. Number of Standards Used: 32 <sup>9/3/94</sup> with zero point RI
3. Type of Curve Fit: QUADRATIC
4. LOD: UNDETERMINED
5. LOQ: UNDETERMINED

# ETAMET



**Method Development For Ion Chromatograph**

A. Method Name: CATION 5

Developed By: LEW CRONE  
(Print)

[Signature]  
(Signature)

Reviewed By: Bub Griffin  
(Print)

[Signature]  
(Signature)

B. Objective of Method: ANALYSIS of monovalent cations in a  
High (ppm)  $\text{NaH}_4$  level matrix, similar to  
SLG wet layup conditions. This is a  
loop injection, isocratic method.

C. Instrument Identification: UNIT 2 B100 on-line ABI 2 System 1  
(Unit, System, CIM)

D. Columns: Separator: CS 10  
Guard: N/A  
Concentrator: N/A LOOP INJECTION

E. Suppressor: CMMS II



G. Eluent

Preparation:

36 mL HCl / .25 M DAP

12.0 mLs concentrated Ultrapure HCl and .14 gms  
Fluka DAP to 4 liters DI water.

Flow Rate:

1.0

Conductivity:

1.2  $\mu$ S

H. Regenerate:

Preparation:

100 mM TBAOH

50 mLs concentrated TBAOH diluted to 1 liter  
DI water. Spurge with Nitrogen 15 mins  
prior to use. EMPTY OLD REGENERANT

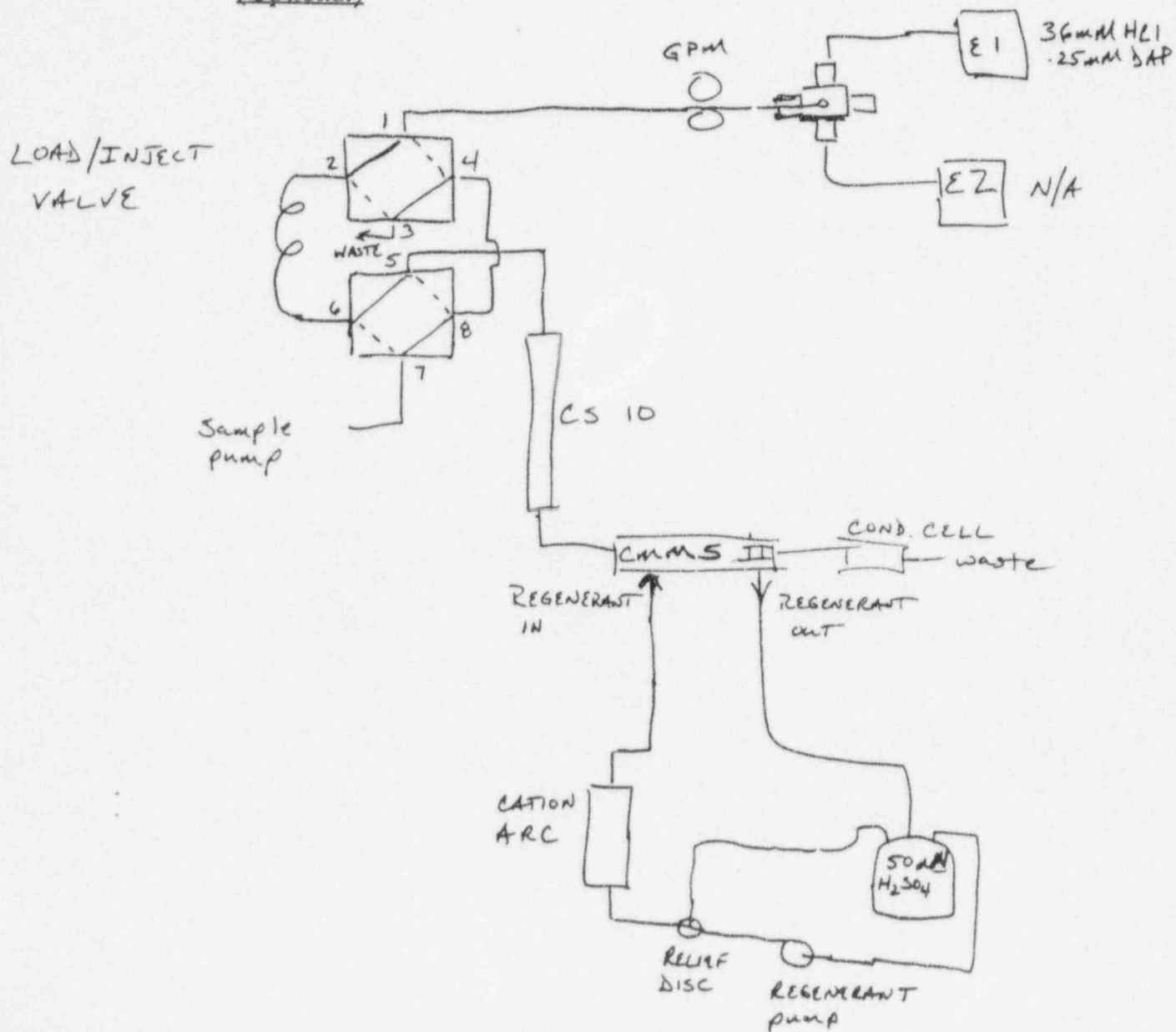
PLACE NEW REGENERANT IN CONTAINER

INSTALL NEW CATION ARC, FLOW BOTTOM  
TO TOP. CONNECT FLUSH LINE TO TOP OF  
CARTRIDGE. PURGE 500 mLs REGENERANT THEN  
NEW CARTRIDGE. REMOVE FLUSH LINE AND  
CONNECT TO CATIONS INLET LINE

Flow Rate:

N/A

F. Plumbing Description/Schematic:  
(Optional)



I. Standards:

Calibration Standards			
Chemical Specie	Concentration 1	Concentration 2*	Concentration 3*
1) SODIUM	.010 ppm	.050 ppm	.100 ppm
2) AMMONIUM	.100 ppm	.500 ppm	1.0 ppm
3) ETHANOLAMINE	1.0 ppm	3.0 ppm	5.0 ppm
4) HYDRAZINE	75.0 ppm	1.25 ppm	150 ppm
5)			
6)			

\*Optional

Control Standards			
Chemical Specie	Concentration 1	Concentration 2*	Concentration 3*
1) SODIUM	.020 ppm		
2) AMMONIUM	.200 ppm		
3) ETHANOLAMINE	2.0 ppm		
4) HYDRAZINE	100 ppm		
5)			
6)			

\*Acceptable deviation should be determined by statistical analysis.

Preparation: HYDRAZINE IS PREPARED FROM HYDRAZINE DICHLORIDE  
SALT. OTHER SPECIES ARE PREPARED THROUGH  
DILUTION OF CONCENTRATED STOCK SOLUTIONS

J. Calibration Data:

1. Range of Method: SEE BELOW
2. Number of Standards Used: 3 (ZERO LISTED AS 1<sup>ST</sup> pt)
3. Type of Curve Fit: QUADRATIC
4. LOD: INCOMPLETE
5. LOQ: INCOMPLETE

RANGES

$\text{Na}^+$  1 ppb - 100 ppb

$\text{NH}_3^+$  20 ppb - 1 ppm

ETA 100 ppb - 5 ppm

$\text{N}_2\text{H}_4$  5 ppm - 150 ppm

-Method Updated: 14:17 on Thu, 09 Jun 1994

Component: Sodium

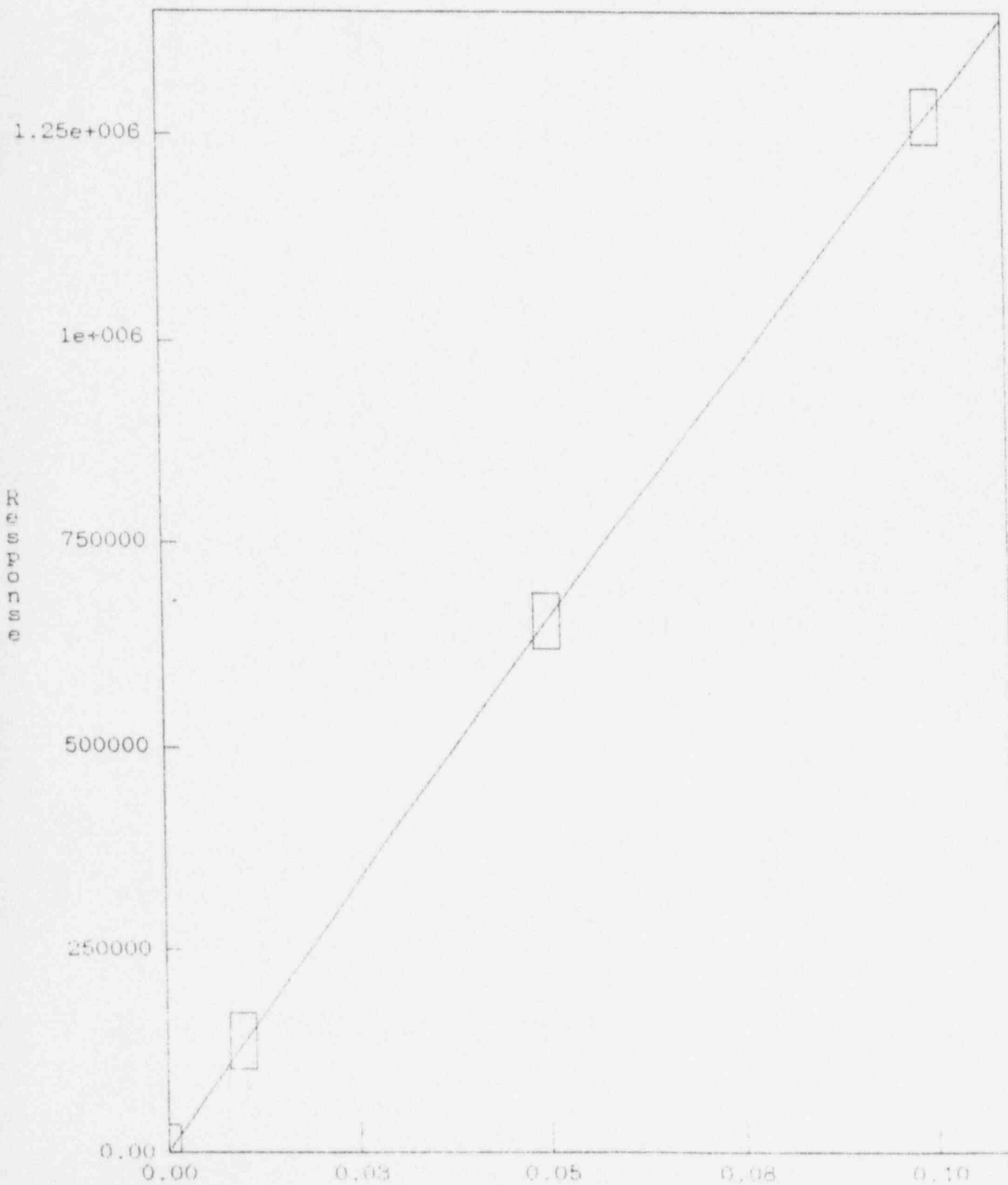
Fit Type: Quadratic

$r^2 = 0.999991$

Amt = (  $3.158047\text{e-}015 * \text{Resp}^2$  ) +  
(  $7.460499\text{e-}008 * \text{Resp}$  ) + -0.0001

Standardization: External

Calibration: Height



Method: C:\DX\METHOD\CATION5.MET

Component: Ammonium

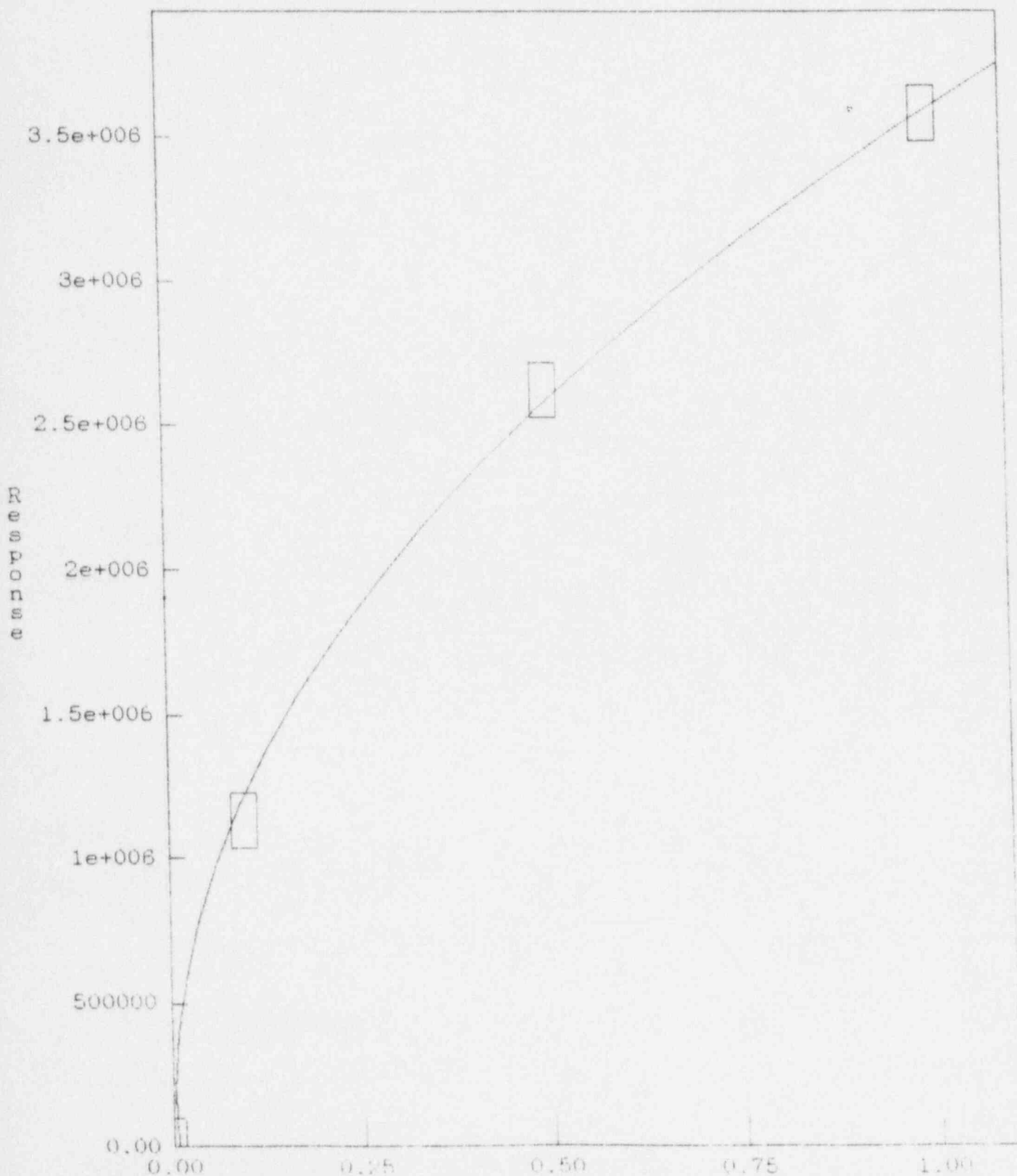
Fit Type: Quadratic

$r^2 = 0.998920$

Amt = (  $8.449920e-014 * Resp^2$  ) +  
(  $-2.671342e-008 * Resp$  ) + 0.0062

Standardization: External

Calibration: Height





Method: C:\DX\METHOD\CATION5.MET

Component: Ethanolamine

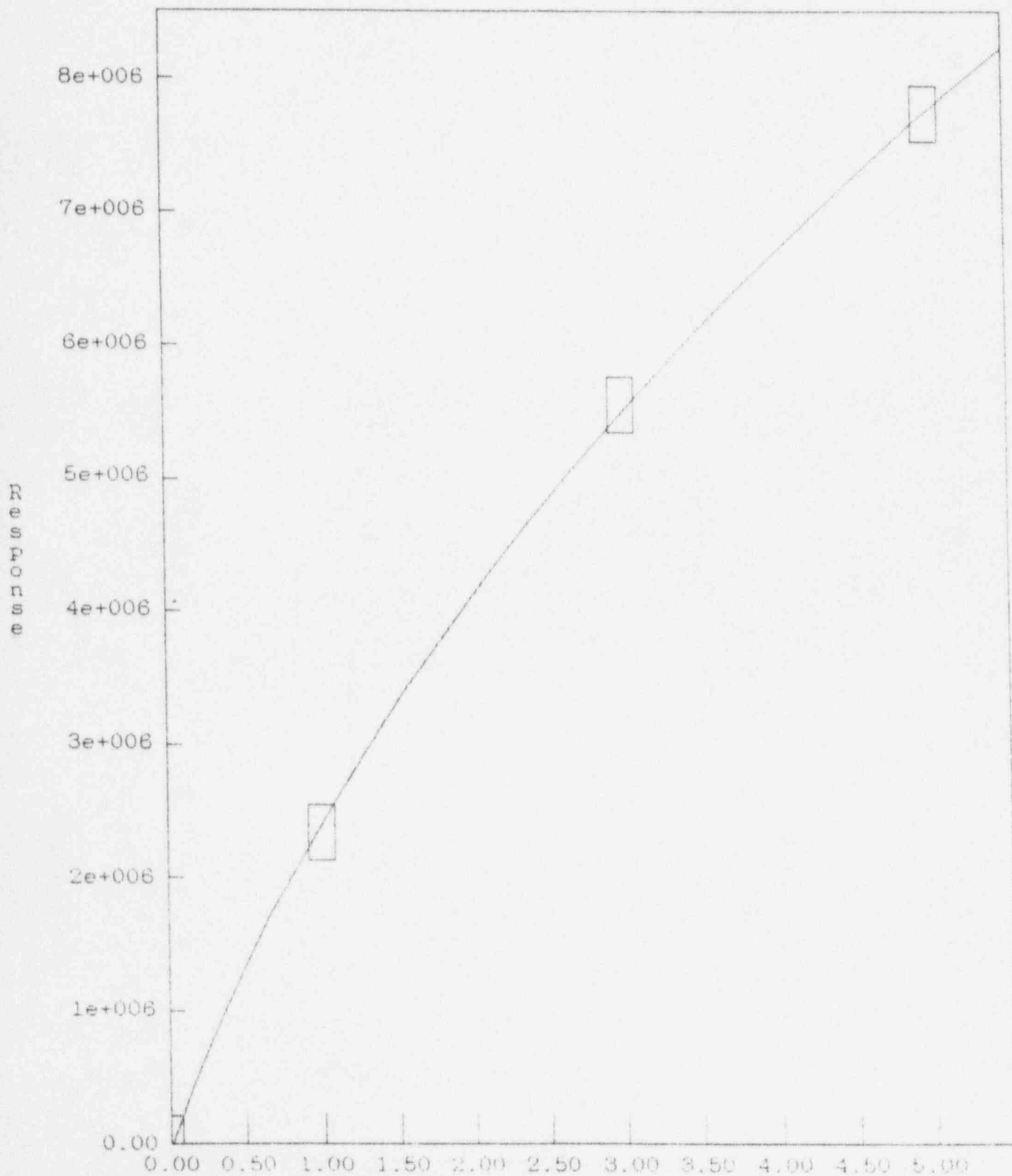
Fit Type: Quadratic

$r^2 = 0.999725$

Amt = (  $4.434428e-014 * Resp^2$  ) +  
(  $2.990569e-007 * Resp$  ) + 0.0164

Standardization: External

Calibration: Height



Method: C:\DX\METHOD\CATION5.MET

Component: Hydrazine

Fit Type: Quadratic

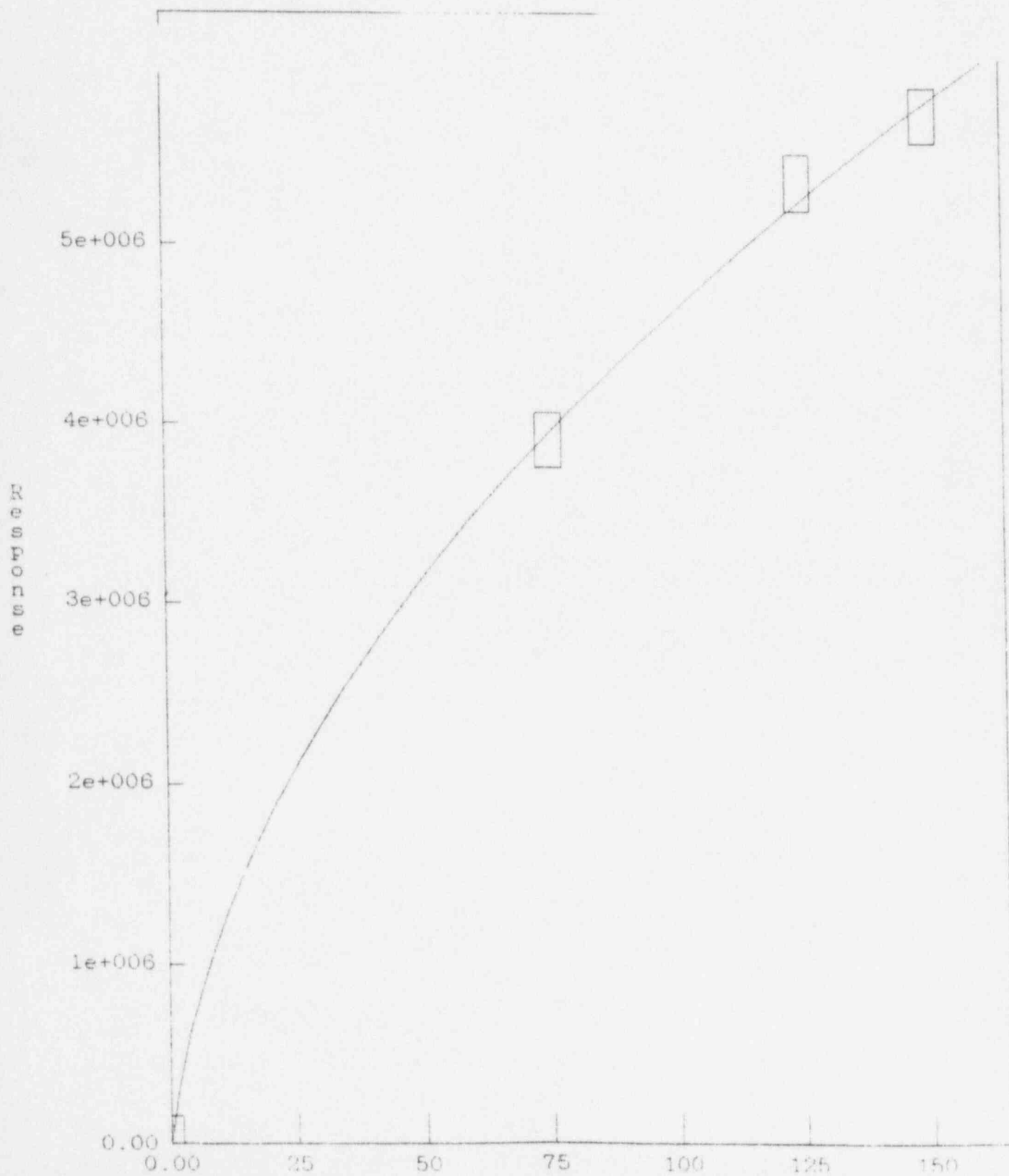
$r^2 = 0.997489$

Amt = (  $3.793548e-012 * Resp^2$  ) +

(  $3.994968e-006 * Resp$  ) + 0.1075

Standardization: External

Calibration: Height



```

=====
Sample Name: CONTROL                               Date: 06/09/1994 14:23:52
Data File  : C:\DX\DATA\CATION5\DATA0061.D01
Method     : C:\DX\METHOD\CATION5.MET
ACI Address: 2 System: 1 Inject#: 1               Detector: CDM-2
Analyst    :                                     Column:
=====

```

```

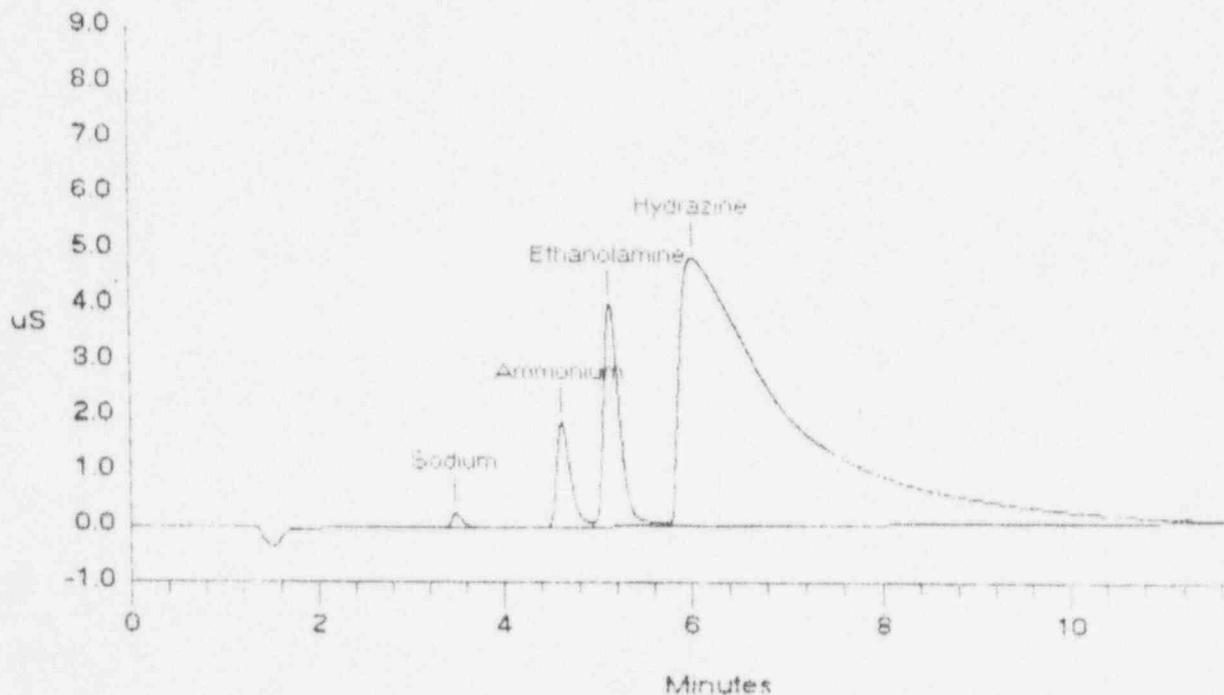
-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          1 3529 5Hz 0.00 11.76          10
-----

```

\*\*\*\*\* Component Report: All Components \*\*\*\*\*

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
			Actual /				
1	3.48	Sodium	.020	0.020	262402	2327176	1 0.00
2	4.62	Ammonium	.200	0.258	1889880	19885931	2 0.00
3	5.13	Ethanolamine	2.0	1.920	3996034	47926058	2 -4.69
4	6.03	Hydrazine	100	109.080	4858884	393765727	2 0.00
Totals				111.276	11007200	463904892	

File: DATA0061.D01 Sample: CONTROL



```

=====
Sample Name: SG1                               Date: 06/14/1994 13:17:57
Data File  : C:\DX\DATA\CATIONS\DATA0401.D01
Method     : C:\DX\METHOD\CATIONS.MET
ACI Address: 2  System: 1  Inject#: 1          Detector: C18-2
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          1  3750  5Hz  0.00 12.50          10
-----

```

\*\*\*\*\* Component Report: All Components \*\*\*\*\*

PK Num	Ret Time	Component Name	Concentration ppm	H	Area	St. Code	Delta
1	3.48	Sodium	0.004	55274	176350	1	0.00
2	4.58	Ammonium	0.175	1579236	16030846	2	0.00
3	5.02	Ethanolamine	15.554	15648004	226498331	3	0.18
4	5.72	Hydrazine	158.477	5956068	498908312	4	0.00
Total			174.210	23238650	741914458		

*File DATA0401.D01 Sample SG1*

